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PATENT
Attorney Docket No. 30811/40225

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Held et al.
Serial No.: 10/795,944
Title: Method For Treating Waste-Activated Sludge Using Electroporation
Filed: March 8th, 2004
Group Art Unit: not yet available
Examiner: not yet available

DECLARATION OF SATYA P. CHAUHAN

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Satya P. Chauhan, hereby state as follows:

1. My name is Dr. Satya P. Chauhan.
2. I am the listed co-inventor of subject matter claimed in U.S. Patent Nos. 6,395,176; 6,491,820; 6,540,919; and 6,709,594 ("the DH2O Patents"). I am also the co-inventor on the above-identified pending application.
3. I am currently employed as a research leader at Battelle Memorial Institute ("Battelle"), where I am responsible for managing multi-sector programs in process and environmental technology. I also lead the commercialization of several Battelle-developed pollution prevention technologies.

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4. I am currently managing three technologies that reduce water and air pollution. One of these technologies uses a pulsed-electric field (PEF) process to reduce the quantity of biological sludges from wastewater treatment plants requiring disposal.

RELATED WORK EXPERIENCE

5. I have worked at Battelle for 30 years and have led programs in many areas of research including pollution prevention, advanced separations, electrotechnologies, waste treatment, remediation, chemical process development, clean fuel technologies, and minerals processing. Over those 30 years, I have developed extensive technology development experience and have headed groups of scientists and engineers involved in developing processes at laboratory-, bench- and pilot-scale levels, as well as prototyping levels.

6. I have authored or co-authored over 100 publications. I am an inventor on ten (10) United States patents, all within the field of pollution prevention.

7. I have over 20 years experience in conducting or managing advanced separations R&D. This expertise includes flocculation, dewatering, electro-coagulation, electro-osmosis, membranes, selective ion exchange, supercritical fluids processing, electroporation and advanced absorption systems. I also have experience in electrolysis and electroplating systems. For example, I managed a multi-million dollar project of Battelle for the U.S. Air Force that involved identification, development, and demonstration of separations technologies for removing impurities from nickel and chromium plating baths.

8. By virtue of my education, training, and experience, I believe that I am competent to offer opinions about what average practitioners in my field would think about Japanese patent application JP 1-210100 discussed below.

BACKGROUND OF THE TECHNOLOGY

9. Waste-activated sludge (WAS) is produced by municipalities and industrial wastewater treatment facilities throughout the country and is a combination of various bio-solids (e.g., microorganisms such as bacteria or protozoa) and water molecules. The DH2O patents disclose, *inter alia*, techniques to use electroporation to break down cell units and release intracellular dissolved, organic matter, which may be used as a "food" for bacteria, such as via a feedback loop to an originating aeration tank.

10. Electroporation is a phenomenon in which a cell is exposed to high voltage electric field pulses, which destabilize cell membranes, by creating holes and thus increasing permeability. This breakdown leads to the release of intracellular bio-solids and water molecules, and this allows for the use of such materials as a food for an aeration unit. The released materials may be used for biological digestion in aerobic, anoxic, facultative, or anaerobic bioreactors, as well. In any of these examples, the treatment by electroporation may greatly reduce the mass of the sludge requiring disposal.

11. Although electroporation was in use in the food industry, electroporation for sludge treatment was historically quite difficult to achieve—my own work with respect to the DH2O properties involved various testing over a number of years. Based on my experience in the field of waste treatment, research in the field of pulsed-electric field treatment of bio-material historically suffered from a lack of understanding of the physical mechanisms at play in the electrochemical reactions associated with PEF. This research has suffered as well by the inability to measure a systems' reaction to different operating conditions.

12. Electrolysis and arcing, for example, are related phenomena that can hinder electroporation deployment in aerobic and anaerobic digestion systems. Electrolysis

results from the application of high electric fields over a long enough pulse width. If the "on" time (or pulse width) for a high voltage electric field is long enough, individual molecules may be broken down during a PEF treatment, thereby releasing gases during treatment (e.g., hydrogen, oxygen, chlorine, and other gases). Electrolysis is also evidenced by plating, or the collection of metal on the cathode of a PEF system, and corrosion on the anode. A further hazard from electrolysis is arcing, which can damage electrode equipment and, in a worst case scenario, even create an explosion hazard with the gases released during electrolysis. In fact, the amount of arcing increases as more gas is released during electrolysis.

13. As is known in the field of electrochemical engineering, the pulse width, or "on" time, of a high voltage electric field pulse creates electrolysis. The frequency with which that pulse is applied defines the "off" time between pulses. During the "on" time, the high voltage field is applied to cells within the treated material to be treated. The electrodes applying the field act as capacitor plates, storing the applied electric field in the biomass therebetween. If the "on" time of a pulse is long enough, then the electrodes (similar to a capacitor) will completely charge during this "on" time, after which electrolysis can occur. The electrolysis may be evident by cell breakdown and gas release or, in more extreme conditions, by arcing, as discussed above. Attachment A discusses the concept of "capacitance effects" which govern this electrochemical reaction. Short pulses may be used to avoid these capacitance effects, because with short pulses there is insufficient time for enough charge to build up in the system to create an electrochemical reaction. Figure 4.2(c) in Attachment B also illustrates this condition. The short pulses distort (or dampen) the faradic current, j_F , so that the faradic current never reaches the imposed current amplitude, j_i , thus minimizing electrochemical activity. In any event, when looking at whether electrolysis occurs in a system or not, the pulse width of the high voltage applied field is determinative.

"THE KOHO APPLICATION" (JP 1-210100)

14. I have reviewed an English translation of Japanese patent application, JP 1-210100 (Attachment C, hereinafter "The Koho application"), which was cited in Japanese Patent Application No. JP 2002-509028, which is a foreign counterpart to U.S. Patent 6,395,176.

15. The Koho application relays a method for anaerobic digestion of sludge using a high-voltage pulse applied to the sludge. The Koho Application states:

If a high-voltage pulse is applied to the sludge, a major portion of the electric energy is applied to the cell walls of microorganisms present in the sludge; this energy is converted into a membrane compression energy, and the cell walls are destructured.

16. The Koho application describes specific operating conditions for its high-voltage pulse sludge treatment. Electric field intensity is to be between 5-50 kV/cm. Pulse widths are to be greater than or equal to 100 μ s ("not less than 100 μ sec"), and preferably 200-1000 μ s or higher.

A working example is provided:

| | |
|---------------------------|-------------|
| Electric Field Intensity: | 25 kV/cm |
| Pulse Width: | 500 μ s |
| Pulse Spacing: | .1 s |
| Pulse Frequency: | 10 Hz |
| Treatment Time: | 20 minutes. |

17. The Koho application acknowledges that electrolysis can occur under certain conditions of high-voltage electric fields. Yet, the Koho application fails to recognize any of the hazardous problems with electrolysis (see, ¶12 above), but instead states that electrolysis "is meaningless from the standpoint of destructing cell walls." The Koho

application suggests that electrolysis should only be avoided to prevent increased power consumption. The Koho application does not recognize the many detrimental affects that can occur with substantial electrolysis in a treatment system.

**THE DISCLOSED RANGES IN THE KOHO APPLICATION WILL CREATE
SUBSTANTIAL AMOUNTS OF ELECTROLYSIS**

18. Having reviewed the Koho Application, my opinion is that the application would not enable one of ordinary skill in the art to build or derive sludge treatment systems like those described in the DH2O Patents. I base this opinion on the operating ranges described in the Koho Application, in particular the disclosed pulse width ranges, which would result in electrolysis contrary to what is suggested.

19. Although Koho apparently believed that electrolysis could be avoided by pulses of 100 μ s or greater, in fact, in my opinion that is not the case. A substantial amount of electrolysis is expected to occur under conditions described by the Koho Application. Data more recent than that of the 1988 Koho timeframe, including data from tests that I am familiar with and data from tests that I designed and monitored (*see, e.g., ¶20*), establishes that extensive electrochemical activity will occur at the 100 μ s and above pulse widths to which the Koho application is directed.

20. In 2002, for example, Battelle conducted tests at a municipal waste treatment facility in Lancaster, Ohio. Using a Pulsed Electric Field system, like that disclosed in the DH2O Patents, Battelle processed waste sludge for 23 days. For the tests, the pulse widths ranged from 2 to 8 μ s, the voltage was approximately 25 kV/cm, and the frequency was greater than 1000 Hz (*e.g., 2000-3000 Hz*).¹ During these tests, the PEF

¹ The applied electric field was the same as that of the Koho Application, although the overall frequency was different. Yet, as described in ¶13 and the appendices referenced therein, signal frequency does not affect the amount of electrolysis; instead, the pulse width affects the amount of electrolysis and arcing.

system began to experience unstable operation at pulse widths of 8 μ s and higher. The unstable operation was system arcing attributable to electrolysis. At pulse widths of 8 μ s and above, bubbling was evident in the sludge thus evidencing gas formation from electrolysis. Because the PEF system was designed to protect electronics in the event of detrimental amounts of arcing, the PEF unit would periodically shut itself down when the testing went above approximately 8-9 μ s to make sure that excessive arcing levels were not reached. PEF operation in the longer pulse width range became unstable due to an increased tendency for arcing. At shorter pulse widths, however, gas formation and arcing was not observed.

21. Thus, based on the general knowledge in the field of electrochemical technology and from my observations based on the above-referenced tests which showed electrolysis beginning to show at pulse widths much smaller than those disclosed by the Koho Application, substantial amounts of electrolysis would result when using the 100 μ s and above pulse widths suggested by the Koho Application. These pulse widths are substantially longer than those of the Battelle tests at the Lancaster facility, which resulted in some electrolysis. Further, as is well known, once electrolysis has occurred, increasing the pulse width of the applied electric field will exponentially increase the rate of arcing because of the increased gas evolution (or release) due to electrolysis. As the rate of arcing increases substantial amounts of arcing will occur resulting in a high likelihood of equipment damage or gas explosion. The pulse widths described in the Koho Application would not only result in electrolysis, but in substantial and excessive arcing from this electrolysis. This is in contrast to what is suggested by the Koho Application.

22. Furthermore, anyone reading the Koho Application at or around the time of the filing of the first DH2O Patent would have understood that the long pulse widths described in the Koho Application would result in substantial amounts of electrolysis.

Furthermore, the Koho Application would have only motivated someone to form a PEF sludge treatment system that operated in a range that resulted in substantial electrolysis.

23. As the Koho Application is limited to 100 μ s and above pulse widths, the Koho Application describes a system that will result in substantial electrolysis, whereas the DH2O patents describe ranges and systems that would not result in substantial electrolysis, if any.

CERTIFICATION AND OATH

24. I hereby declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements are punishable by fine or imprisonment, or both, under § 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: June 15, 2004

By

Satya P. Chauhan
Satya P. Chauhan

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